REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

12

1. AGENCY USE ONLY (Leave blank)

2. REPORT DATE
1/21/94

3. REPORT TYPE AND DATES COVERED
Final Report 1/1/89 - 12/31/93

4. TITLE AND SUBTITLE
The Effects of Interstitial Elements on the Phase Stability and Mechanical Behavior of Intermetallics

5. FUNDING NUMBERS
N00014-89-J-1503
orin 005-04
N66005

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8. PERFORMING ORGANIZATION REPORT NUMBER

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Arlington, VA 22217-5000

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1960 Kenny Road
Columbus, OH 43210-1063

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Unlimited distribution

12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

This document constitutes the final report for ONR sponsored research entitled "The Effects of Interstitial Elements on the Phase Stability and Mechanical Behavior of Intermetallics". Alloying with interstitial elements O, N, or C induced structural changes from the A15 crystal structure to the ordered fcc $L1_2$ or $L'1_2$ structures in 16 alloy systems. The $L1_2$ or $L'1_2$ structures exhibited higher fracture toughness values than those of the original A15 structure compounds.

14. SUBJECT TERMS Intermetallic compounds, interstitial elements, structural changes, hardness, fracture toughness

15. NUMBER OF PAGES

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	UL

Final Report

Grant N00014–89–J–1503

THE EFFECTS OF INTERSTITIAL ELEMENTS ON THE PHASE STABILITY AND MECHANICAL BEHAVIOR OF INTERMETALLICS

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January 25, 1994

Final Report for Period January 1, 1989–December 31, 1993

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A-1	

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FINAL REPORT

Introduction — Description of Research Goals

The objective of the research was to explore the potential for mechanical behavior enhancement by structure modification of certain refractory metal-based high melting temperature intermetallics. Three approaches were used toward this end. The first comprised an experimental investigation of the phase-stability, crystal structure, and microstructure of selected intermetallic compounds as a function of interstitial element additions (eg. O, C, N, B). The second involved the prediction of the stabilization of close-packed phases — such as the Ll_2 or the $L'l_2$ — induced by interstitial alloying in complex crystal structures (eg. A15) in high temperature intermetallics. Here, the development of empirical correlations utilized such elemental parameters as atomic radii, elecronegativity, and valence electron number. The third consisted of a study of the mechanical behavior, particularly fracture toughness, of interstitially stabilized structures.

Major Accomplishments

1) The survey explored over twenty intermetallic alloy A_3B compounds — mostly with the complex Cr_3Si (A15) crystal structure. It was found that no less than sixteen of these were capable of being transformed into the simpler ordered fcc Ll_2 or $L'l_2$ structure by interstitial alloying.

2) In most material systems, structure stability diagrams were found to classify or predict the expected crystal structure; however, in some instances, there was disagreement between the predicted and the observed crystal structure. In the cases where there was disagreement, the violations were in such a way that they may actually represent small modifications to the maps.

3) The fracture toughness of selected intermetallic compounds was

estimated using a method that involves the measurement of crack lengths emanating from the corners the Vickers indenter impressions (method of Marion — ASTM Special Techniques Publication No. 678). The toughness values of the transformed fcc structure alloys were observed to be superior those of the untransformed base alloys. In some cases, the toughness value, K_{IC}, more than doubled after complete transformation. In the same instances, diamond point microhardness measurements were determined and found to decrease in most cases and increase in very few cases — typically involving the interstitial alloying of carbon.

4) The application of fractal geometry measurements to the evaluation of fracture toughness of brittle intermetallics was examined. Boundary fractal dimensions of planar sections through the fracture surface of the intermetallic compound V_3Au + O were determined at varying oxygen percentages — up to 20 atomic percent. It was shown that the mean measured fractal dimension maintained a consistent relationship with the fracture toughness values obtained using the indentation technique. Furthermore, it was observed that this behavior agrees with data from the literature involving two very different families of brittle materials: polycrystalline alumina and glass-ceramics. General agreement was observed despite the large differences in grain size, modulus, and fracture energy of the materials and suggests the possibility of a new method of surveying fracture toughness where conventional techniques may not be applicable.

5) Hot hardness behavior of the A15 V_3Au and $L'1_2$ V_3AuO were measured by Dr. Steve Bruemmer at PNL. The magnitude of the hardness for V_3AuO crosses and exceeds that for V_3Au at temperatures above 650°C. This suggests that the fcc-like $L'1_2$ perovskite phase is stronger at elevated temperatures than the A15 phase.

6) The dependence of diamond point hardness on grain size was determined for the V_3Au A15 base alloy and the V_3AuO $L'1_2$ perovskite alloy. In both cases the behavior was fit to a Hall-Petch relationship.

7) A miniature disk-bend test (MDBT) apparatus was fabricated and calibrated to known samples. The device, used in conjunction with a tensile machine, is capable of yielding the stress vs. strain relationships of small thin disks.

8) Miniature bend-bars of the V_3Au intermetallic alloy (base A15 and transformed $L'1_2$) were produced. The bend specimens were clamped in compression, then buckled until fracture occurred. Fracture strains obtained in this manner were in qualitative agreement with fracture toughness measurements made using the indentation technique.

9) TEM studies were conducted to observe the microstructural behavior of low oxygen (0 – 1 atomic percent) content V_3Au alloys. In this regime, the hardness is observed to increase sharply. TEM revealed that this effect is due to the initial formation of the $L'1_2$ perovskite phase as a very fine (less than one micron) dispersion at the grain boundaries.

<u>Publications</u>

1. M. A. Kassem, Y. Fahmy and C. C. Koch, "The Influence of Interstitial Alloying Elements on the Phase Stability and Fracture Toughness of V_3Au", Materials Research Society Symposium Proceedings, Vol. 186, 369–374 (1991).

2. M. A. Kassem and C. C. Koch, "Effect of Interstitials on the Phase Stability of Selected Intermetallics", Materials Research Society Symposium Proceedings, Vol. 213, 801–806 (1991).

3. Y. Fahmy, J. C. Russ and C. C. Koch, "Application of Fractal Geometry Measurements to the Evaluation of Fracture Toughness of Brittle Materials", Journal of Materials Research, Vol. 6, No. 9, 1856–1861 (1991).
4. Y. Fahmy, C. T. Benfield and C. C. Koch, "The Effects of Interstitial Elements on the Phase Stability and Mechanical Behavior of Selected Intermetallics", Materials Science and Engineering, A170, 19–27 (1993).

Dissertations

1. Magdy A. Kassem, "Effect of Interstitial Elements on Phase Stability of Selected Intermetallics". Doctor of Philosophy Thesis, December 1990.
2. Yusef Fahmy, "The Influence of Interstitial Elements on the Mechanical Behavior of the V_3Au Intermetallic Compound". Master of Science Thesis, May 1992.
3. Timothy C. Benfield, "The Influence of Interstitial Elements on the Phase and Mechanical Behavior of Selected Intermetallic Compounds". Master of Materials Science and Engineering, December 1992.
4. Yusef Fahmy, "Modification of the Mechanical Behavior in A15 Intermetallic Compounds by Structural Changes Induced by Interstitial Alloying". Doctor of Philosophy, in preparation, 1994.

Invited Presentations

1. C. C. Koch, "The Effects of Interstitial Alloying Elements on the Phase Stability and Mechanical Behavior of Intermetallics", General Electric Corporate Research Laboratory, Schenectady, New York, February 17, 1989.
2. C. C. Koch, "Stabilization of $AuCu_3$ and Perovskite Structure Types by Interstitial Carbon, Nitrogen, and Oxygen", TMS Hume-Rothery Memorial Symposium, New Orleans, LA, February 18, 1991.

3. C. C. Koch, "The Effects of Interstitial Elements on the Phase Stability and Mechanical Behavior of Intermetallics", Industrial Technology Research Institute, Materials Research Laboratories, Hsinchu, Taiwan, Republic of China, May 17, 1991.

4. C. C. Koch, invited presentations at ONR review meetings of "Ordered Intermetallics" Program contractors.

    a) Arlington, VA, May 12, 1989

    b) Arlington, VA, May 17, 1990

    c) Annapolis, MD, June 24–25, 1991

    d) Annapolis, MD, October 14–15, 1992.